Energy, Mines and Resources Canada | Énergie, Mines et Ressources Canada

CANMET

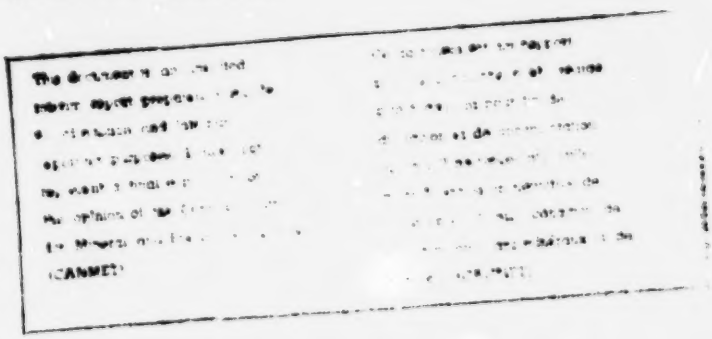
Canada Centre for Mineral and Energy Technology | Centre canadien de la technologie des minéraux et de l'énergie

DEVELOPMENT OF A METHOD FOR THE STUDY OF THE KINETICS OF CHLORINATION OF FERROUS CHLORIDE LEACH SOLUTIONS

K.G. Tan and P.L. Bedard
Metallurgical Chemistry Section
Extractive Metallurgy Laboratory

November 1985

Project: MRP-3.0.52.02
Chloride Processes for Sulphide Ore Treatment



MINERALS RESEARCH PROGRAM
MINERAL SCIENCES LABORATORIES
DIVISION REPORT MRP/MSL 85-137 (TR)

Crown Copyrights Reserved

MICROMEDIA

JAN 21 1988

24/77

DEVELOPMENT OF A METHOD FOR THE STUDY OF THE KINETICS OF CHLORINATION OF FERROUS CHLORIDE LEACH SOLUTIONS

by

K. G. Tan* and P. L. Bedard**

ABSTRACT

The kinetics of regeneration of ferrous chloride leach solutions by chlorination are to be explored for process design purposes. A 25 x 2 cm packed column with appropriate accessories was set up, and test runs were conducted in a co-current operational mode at different temperatures and reagent flow rates. The results indicate that reliable estimates of the mass transfer rates at different points along the column can be evaluated and that a higher column loading is feasible at lower temperatures.

Basic design considerations, preliminary results and a data evaluation method are discussed as a basis for the mini-plant testing of CANMET's ferric chloride leach process for complex sulphide concentrates. Further improvements to the apparatus and to the test procedure are required for more accurate kinetic measurements. Such work would provide useful fundamental insight into the mechanisms involved, and also may be applicable to the study of other fast reactions.

---

*Research Scientist and **Technician, Metallurgical Chemistry Section, Mineral Sciences Laboratories, CANMET, Energy, Mines and Resources Canada, 555 Booth Street, Ottawa, Ontario K1A 0G1.

ÉLABORATION D'UNE MÉTHODE POUR ÉTUDIER LE COMPORTEMENT CINÉTIQUE DU PROCÉDÉ DE CHLORURATION DES SOLUTIONS DE LIXIVIATION DE CHLORURE FERREUX

par

K.G. Tan* et P.L. Bédard**

RÉSUMÉ

On doit étudier le comportement cinétique de la régénération des solutions de lixiviation de chlorure ferreux par le procédé de chloruration afin de faire le calcul fondamental d'une installation. Une colonne de garnissage de 25 x 2 cm avec les accessoires appropriés a été montée, et des essais témoins ont été faits suivant un mode opérationnel en courant continu à des températures et des débits de réactifs différents. Les résultats d'essais ont montré qu'un calcul approximatif fiable des vitesses du transfert de masse fait à différents points au long de la colonne peut être déterminé et qu'un chargement plus élevé de la colonne est réalisable à des températures plus basses.

Les observations sur la conception de base de l'installation, les résultats préliminaires et la méthode d'évaluation des données ont été traités dans ce rapport en tant que base aux essais de la mini-installation du procédé de lixiviation au chlorure ferrique pour les concentrés de sulfures complexes de CANMET. L'appareillage et les procédures d'essais nécessiteront plus de perfectionnement afin d'obtenir des mesures plus précises du comportement cinétique. Un tel travail fournirait un aperçu fondamental et utile des mécanismes en jeu et pourrait également servir à l'étude d'autres réactions rapides.

---

*Chercheur scientifique et **Technicien, Section de la chimie métallurgique, Laboratoires des sciences minérales, CANMET, Énergie, Mines et Ressources Canada, 555, rue Booth, Ottawa (Ontario) K1A 0G1.

INTRODUCTION

In an earlier study (1), the development of a mini-plant chlorinator for the regeneration ferrous chloride leach solutions was discussed. The design was tested with typical "raffinates" containing about 1 M $FeCl_2$, 3 M NaCl and 0.5 M HCl, with various levels of other impurities present (lead, zinc, copper, gypsum). Although satisfactory operation at different temperatures and flow rates up to 1 L/min was achieved, it was concluded that the generation of useful kinetic and engineering data would require an improved laboratory set up with positive control of all process parameters. Such an undertaking would benefit not only the chloride process development work, but would also provide a convenient means of studying other fast reactions in a dynamic mode.

The earlier design and estimating procedures (1) were used to construct a 2 cm diameter column, packed with 0.6 cm glass Raschig rings and capable of treating up to 100 mL/min of raffinate. Different accessories and equipment configurations were also evaluated in this study. While some improvements are still required for accurate kinetic measurements, an adequate working design has been achieved and a reliable data evaluation method has been developed. The purpose of this report is to document the progress to date, and to present some of the results which may be useful for ongoing mini-piloting work elsewhere (2).

EXPERIMENTAL

The analytical approximation for gas film control (1) predicted a column length of 21 cm for a target flow of 100 mL/min of raffinate. The equipment configuration was modified on a step-by-step basis, and the final working design is shown in Figure 1.

A 25 cm length of PVC pipe (2 cm inner diameter) was fitted with a bottom end cap which held a fritted glass sparger and the solution inlet tube. Two sampling valves and a thermocouple probe were inserted through the pipe wall at the indicated locations, and the column was packed to the top with 0.6 cm glass Raschig rings. A single outlet tube was mounted through the upper end cap, and the column was completely covered with a layer of glass wool/aluminium foil insulation. The outlet was directly connected to an outlet sampling valve and a small ORP (oxidation/reduction potential) measuring cell which could serve as a gas/liquid separator as well. To minimize chlorine emissions, however, the overflow was led into a larger disengagement vessel, as shown. Two matched peristaltic pumps were required and all connections were secured with epoxy glue or clamps.

The major features of this design are the elimination of dead-space in the column to improve the response time to parametric changes, and also the removal of the precontactor to ensure a constant Fe^{2+} inlet concentration. Feed solution was pumped via a calibrated rotameter and a preheating flask into the column. Chlorine was also metered into the column via a calibrated rotameter with a glass float and Monel body. Feed, inlet and column temperatures were monitored using a manual switching station connected to a dual pen recorder. The outlet ORP was also monitored with a combination saturated calomel electrode (SCE) and the measured voltages were recorded. Instrument readings, sampling and parametric changes were scheduled on a regular basis throughout the runs. To minimize upsets during sampling, small samples (~5 mL) were carefully taken in an upstream direction; i.e., first the product, next the outlet, then valve 2, and finally valve 1. The Fe^{2+} content was determined by titration with dichromate, and total iron, sodium, lead, zinc, copper and calcium were measured by atomic absorption spectrophotometry.

To convert product solution into feed for the next run, an automatic iron reduction column was operated. This consisted of a 4 L glass cylinder packed with steel wool that overflowed into a smaller cylinder packed with glass wool as a prefilter. Dilution with water by 50% also took place at the bottom inlet of the reducer to enhance the rate of conversion and to yield the appropriate iron concentration for the next run. Additional adjustments (polish filtration, analytical determinations and reagent additions) were carried out before recycling the final solution.

RESULTS

The bulk of the testwork involved modifications to the equipment, and the results of these preliminary tests are summarized in Figure 2. Although some correlation may be established between the percentage of the ferrous conversion (bottom part of the figure) and the relative gas and liquid flow rates (top part of the figure), the column response was very sluggish and insensitive to major parametric changes. This may be attributed to the initial design (1) which incorporated a precontactor and a large gas/liquid disengagement space at the top of the column. In addition, high temperature and high pressure runs were problematic because of tube softening, level control and gas flow regulation difficulties. A room temperature run at high pressure (Run 10; not shown) was aborted because the column outlet became completely plugged with debris from the disintegrated sparger and dislodged PVC threads.

Column performance was dramatically improved in Run 11 by eliminating column dead-space and major downstream gas flow restrictions. At a higher inlet temperature (Run 12), plug-up problems due to crystallization were encountered, but insulating the column (Run 13) improved its performance

considerably. Throttling the exit gas flow to the precontactor at room temperature (Run 14) destabilized the operation although high conversion rates were achieved even without the precontactor. The general conclusions from these tests were that an adequate design was achieved for measuring the chlorination kinetics of ferrous chloride leach solutions. High conversion rates were achieved with substoichiometric additions of chlorine and liquid flow rates up to 140 mL/min.

To demonstrate the data evaluation method used, three additional tests using the final apparatus design (Figure 1) are discussed in greater detail. Test 15 (Table 1 and Figure 3) was started with a very small (~10 mL) precontactor cell and a preheated feed solution at 75°C. Heat losses to the pump and rotameter yielded an average column inlet temperature of 55°C. The unstable performance during the first 40 min (Figure 3) was due to pressure control and precontactor level control problems. Both the temperature and ORP profiles stabilized after removing two major gas flow obstructions: a throttle valve and the precontactor sparger. Towards the end of the run, the feed temperature dropped and the precontactor cell was replaced by a 2 L preheater vessel. The ORP trace, disregarding the spikes generated by sampling, is clearly divided into different plateaus corresponding to the changes in gas and liquid flows. The ORP dropped with higher liquid flow rates, despite the excess chlorine supplied under these conditions.

Test 16 (Table 2 and Figure 4) displays the excellent response to parametric changes for an inlet temperature between 25 and 28°C. Since the ORP readings are not corrected for temperature, they are lower than in the previous run although they actually correspond to higher conversions at substoichiometric chlorine additions. The optimum column loading seems to be established at a liquid flow of 100 mL/min, because more chlorine is

required to maintain a comparable ORP at higher liquid flows and because no appreciable improvement is gained at 70 mL/min.

Test 17 (Table 3 and Figure 5) demonstrates some of the remaining difficulties associated with elevated temperatures and pressures. The target temperature of 50°C was not easily maintained by the preheater, and the temperature and ORP profiles are much less regular than in the previous run. Plugging and tube softening problems led to difficulties in level control, necessitating a shutdown during the middle of the run. The test results, however, still display distinct variations in product ORP in response to step changes in gas and liquid flow rates. The optimum liquid flow now appears to be below 80 mL/min, since a high ORP is also obtained at 60 mL/min with much lower chlorine additions. A more quantitative evaluation of the results in terms of applicable mass transfer models is presented in the next section.

DISCUSSION

Earlier design considerations (1) suggested that the reaction studied is gas film controlled, and that the required column length may be calculated from equations by Onda et al. (3). For a 2 cm diameter column packed with 0.6 cm Raschig rings, the mass transfer rate is given by:

$$-r_A'''' = 0.00792\ p_A.\ G^{0.7} \qquad \text{(mol/L.min)} \qquad (1)$$

Conversions at different sampling locations may then be calculated in an iterative manner according to:

$$dG/V_0 = L.dC_B/b = -r_A''''.\ dV_R \qquad \text{(mol/min)} \qquad (2)$$

In these equations, the nomenclature of Reference (1) has been retained:

P_A = Cl_2-partial pressure (kPa); L, G = liquid and gas flow rates (L/min); V_0 = ideal gas volume (STP) = 22.412 (L/mol); C_B = ferrous concentration (mol/L); b = L/G stoichiometric factor = 2; and V_R = reactor volume (L).

Table 4 shows that this method yielded acceptable values only for the outlet Fe^{2+} concentrations, whereas the agreement with the observed data at the first sampling point of the column was very poor. The success of the preliminary design calculations appears to be fortuitous although the concept of gas film control can be utilized to derive a better fit to the experimental data, as outlined below.

The residual gas flow rates (G) are estimated from the measured Fe^{2+} concentrations according to equation 2, and the appropriate reactor volumes are calculated from the column diameter and the distance of each sampling point from the top of the chlorine sparger. The product sampling point was arbitrarily placed at a 35 cm column length (110 mL reactor volume). For the evaluation of $-r_A''''$ from equation 2, the gas flow rates are plotted as a function of V_R (Figure 6), and tangents (dG/dV_R) are obtained at each data point (Table 5). To derive the dependence of these values on G, several plots with different coordinate systems were constructed, of which the semi-logarithmic graph (Figure 7) displayed the most linear behaviour. The scatter of the data is reasonable considering that the variations in gas flow rates (±10 mL/min) during steady state operation can affect the measured concentrations by up to 25%. More accurate curve fitting methods may be applied when better control of temperature and gas flow rate is achieved. Figure 7 and Table 6 show that an acceptable fit to most of the observed data is obtained by expressing the mass transfer rate as:

$$\log(-r_A'''') = 1.6\ G - 1 \qquad (3)$$

As expected, the column performance is primarily affected by the total flow rate (G+L) and the actual column loading ($L.C_B$). The former determines the retention time, and is initially dominated by the gas flow rate which diminishes rapidly along the column. The implicitly included gas/liquid stoichiometric ratio is also determined by the initial ferrous flow or column loading. Thus, high flow rates tend to yield lower observed conversion rates ($d>0$), whereas low loading enhances the actual performance over that predicted by equation 3 ($d<0$).

The results also confirm that the overall performance is better at room temperature (Run 16), where up to 8 g Fe^{2+}/min was adequately treated. Corresponding loading limits at inlet temperatures of 55°C (Run 15) and 45°C (Run 17) were only about 7 g Fe^{2+}/min. The gas film control model always predicts convergence of the column output levels for the range of flow rates and gas/liquid stoichiometries tested. Fluid dynamics (flooding) and temperature effects are not expressly incorporated in the empirical model, but may be quantified when more accurate measurements are made. Major improvements scheduled are the use of an electronic mass flow controller to meter the chlorine, a data-logger to record the process data continuously, a longer column with several sampling valves to obtain better profiles, more accurate liquid metering pumps, and a proportional controller to maintain a constant inlet temperature.

CONCLUSIONS

Progress has been made in the development of an integrated system for measuring the kinetics of very fast reactions in a dynamic mode and on a laboratory scale. Observations confirm the feasibility of the chlorination of $FeCl_2$ solutions, especially at lower inlet temperatures and pressures,

and some refinements in the gas film control model for calculating the reactor dimensions were achieved. Consequently, more confidence can be placed on the proposed equipment and flowsheet alternatives for regenerating the spent iron chloride leach solution (1) although integrated piloting on a mini-plant scale must still be conducted to provide closed mass balances for a continuous operation.

ACKNOWLEDGEMENTS

Valuable assistance in equipment design was provided by K. Bartels, and helpful suggestions were received from C. Hamer, R. Molnar and L. Wilson.

REFERENCES

1. Tan, K.G. and Bedard, P.L. "The Development of a Mini Plant Chlorinator for the Oxidation of Ferrous Chloride Solutions"; Division Report MRP/MSL 85-29 (TR); CANMET, Energy, Mines and Resources Canada; Ottawa; 1985.

2. Lucas, B.H. and Shimano, D.Y. "Ferric Chloride Leach Process: Progress Report"; Division Report MRP/MSL 83-2 (CF); CANMET, Energy, Mines and Resources Canada; Ottawa; 1983.

3. Onda, K., Takeuchi, H. and Okumoto, Y. Equations 18-55 and 18-68 in "Chemical Engineer's Handbook"; Perry, R.H. and Chilton, C.H. editors; 5th edition; McGraw-Hill; New York; 1973.

Table 1. Operating Data for Run 15

Run Time (min)	Rotameter Readings L	G	Temperatures (Centigrade) T0	T1	T2	Outlet ORP (mV SCE)	Events
0	80	60	75	30	?	?	start, 155 kPa
5	80	50	75	40	48	537	level upset
25	80	60	74	50	42	532	pressure up
35	80	75	74	51	57	560	free gas flow
45	80	75	74	53	58	558	sample 1, decr. G
50	80	60	75	53	57	541	
55	80	60	75	53	57	540	
60	80	60	75	53	57	539	sample 2, incr. L&G
65	115	85	76	59	63	530	feed heater off
70	115	85	74	59	64	527	
72	115	85	72	58	63	526	sample 3, incr. L&G
75	130	110	68	57	62	534	precontactor out
85	130	110	38	56	52	510	preheater in
90	130	110	32	51	53	518	sample 4, incr. L&G
95	150	120	-	-	50	510	
100	150	130	-	-	50	510	end

T0 = feed; T1 = precontactor; T2 = outlet temperature

Sample #	Flows (L/min) L	G	Fe^{2+} Concentrations (g/L) Inlet	Valve#1	Valve#2	Outlet	Product
15-1	0.070	0.89	58.8	26.1	13.1	-	2.0
15-2	0.070	0.84	60.1	28.8	17.6	-	3.2
15-3	0.105	1.35	64.1	35.3	19.6	-	7.8
15-4	0.125	1.61	64.1	35.3	24.8	-	11.8

Table 2. Operating Data for Run 16

Run Time (min)	Rotameter Readings L	G	Temperature (Centigrade) T1	T2	Outlet ORP (mV SCE)	Events
0	120	70	29	?	?	start
15	120	70	27	38	480	
25	120	70	25	38	478	
30	120	70	25	38	475	sample 1, incr. G
32	120	95	25	40	494	
35	120	95	26	41	497	increase G
38	120	120	26	42	506	
45	120	120	27	43	515	
50	120	120	27	44	519	sample 2, decr. L&G
55	80	60	27	44	535	decrease G
60	80	50	27	45	526	
65	80	50	27	44	524	
80	80	50	27	44	523	
90	80	50	26	43	523	sample 3, incr. L&G
95	140	130	25	40	496	
110	140	130	28	42	498	
115	140	130	28	42	498	sample 4, decr. L&G
125	110	105	27	44	523	
135	110	105	27	44	521	
145	110	105	26	44	517	sample 5, decr. G
150	110	75	26	41	494	
160	110	75	27	41	493	
170	110	75	28	42	493	
190	110	75	28	42	494	sample 6, end

T1 = inlet; T2 = outlet temperature

Sample #	Flows (L/min) L	G	Fe^{2+} Concentrations (g/L) Inlet	Valve#1	Valve#2	Outlet	Product
16-1	0.115	1.26	69.5	40.7	23.7	17.5	?
16-2	0.115	1.47	69.5	35.6	16.9	8.5	5.6
16-3	0.070	0.92	69.5	28.2	14.1	6.2	4.0
16-4	0.140	1.67	69.5	39.0	21.5	16.7	10.2
16-5	0.100	1.31	69.5	28.8	14.1	5.6	4.0
16-6	0.100	1.19	69.5	32.8	23.7	18.1	10.2

Table 3. Operating Data for Run 17

Run Time (min)	Rotameter Readings L	G	Temperature (Centigrades) T1	T2	Outlet Pressure (kPa)	Outlet ORP (mV SCE)	Events
0	90	90	44	?	?	?	start
10	90	90	38	50	115	558	
15	90	90	39	50	115	564	
25	90	90	48	56	130	578	
30	90	90	45	55	130	585	sample 1, decr. G
35	90	70	42	53	145	568	
40	90	70	39	49	145	558	
55	90	70	45	54	145	553	sample 2, level up
65	90	70	45	53	145	550	level upset
70	-	-	-	-	-	-	stop, change pump
100	130	130	48	47	145	547	restart
105	130	130	42	50	145	542	
110	130	130	40	49	145	538	
115	130	130	40	50	145	532	
120	130	130	40	50	145	530	
125	130	130	39	48	145	528	sample 3, decr. L&G
135	70	50	40	49	170	575	
140	70	50	44	51	155	560	
145	70	50	47	54	155	565	
150	70	50	48	55	155	565	sample 4, incr. L&G
155	110	80	47	55	170	537	
160	110	80	44	52	170	530	
180	110	80	46	52	170	530	end

T1 = inlet; T2 = outlet temperature

Sample #	Flows (L/min) L	G	Fe^{2+} Concentrations (g/L) Inlet	Valve#1	Valve#2	Outlet	Product
17-1	0.080	1.08	58.5	22.6	8.5	2.3	0
17-2	0.080	0.87	58.5	33.9	19.2	10.2	4.5
17-3	0.125	1.55	58.5	19.8	11.3	-	2.3
17-4	0.060	0.60	58.5	33.3	18.1	10.2	7.9

Table 4. Evaluation of Experimental Data According to Onda's Model*

Sample #	Calculated and Observed Fe^{2+} Concentrations, (g/L)								
	Valve #1			Valve #2			Outlet		
	Calc.	Obs.	d	Calc.	Obs.	d	Calc.	Obs.	d
15-1	35.8	26.1	- 9.7	5.9	13.1	+ 7.2	-	2.0	+ 2.0
15-2	38.3	28.8	- 9.5	11.8	17.6	+ 5.8	4.5	3.2	- 1.3
15-3	42.7	35.3	- 7.4	13.9	19.6	+ 5.7	3.7	7.8	+ 4.1
15-4	43.6	35.3	- 8.3	15.3	24.8	+ 9.5	4.5	11.8	+ 7.3
16-1	50.9	40.7	-10.2	26.2	23.7	- 2.5	17.7	17.5	- 0.2
16-2	48.7	35.6	-13.1	20.3	16.9	- 3.4	9.9	8.5	- 1.4
16-3	45.3	28.2	-17.1	15.1	14.1	- 1.0	6.1	6.2	+ 0.1
16-4	50.7	39.0	-11.7	24.5	21.5	- 3.0	14.5	16.7	+ 2.2
16-5	47.5	28.8	-18.7	18.1	14.1	- 4.0	7.8	5.6	- 2.2
16-6	49.0	32.8	-16.2	22.1	23.7	+ 1.6	13.0	18.1	+ 5.1
17-1	34.8	22.6	-12.2	4.3	8.5	+ 4.2	-	2.3	+ 2.3
17-2	38.2	33.9	- 4.3	13.1	19.2	+ 6.1	5.8	10.2	+ 4.4
17-3	38.5	19.8	-18.7	10.9	11.3	+ 0.4	0.6	2.3	+ 1.7
17-4	38.0	33.3	- 4.7	14.7	18.1	+ 3.4	-	10.2	+10.2

*see equation 1, and references (1) and (3); d = C(obs.) - C(calc.)

Table 5. Mass Transfer Data Calculated from the Concentration Profiles

Sample #	Fe^{2+} Concentration (g/L)				Gas Flow (L/min)				$-r_A''''$ (M/min)		
	0	1	2	3	0	1	2	3	0	1	2
15-1	58.8	26.1	13.1	2.0	0.89	.43	.25	.09	2.28	.42	.19
15-2	60.1	28.8	17.6	3.2	0.84	.40	.25	.05	2.30	.43	.19
15-3	64.1	35.3	19.6	7.8	1.35	.74	.41	.16	7.53	.74	.25
15-4	64.1	35.3	24.8	11.8	1.61	.89	.62	.30	7.18	.83	.29
16-1	69.5	40.7	23.7	17.5	1.26	.60	.20	-	6.24	.87	.27
16-2	69.5	35.6	16.9	8.5	1.47	.69	.26	.06	10.9	.89	.32
16-3	69.5	28.2	14.1	6.2	0.92	.34	.14	.03	4.56	.63	.19
16-4	69.5	39.0	21.5	16.7	1.67	.81	.32	.19	3.63	1.3	.26
16-5	69.5	28.8	14.1	5.6	1.31	.50	.20	.03	8.99	.72	.15
16-6	69.5	32.8	23.7	18.1	1.19	.45	.27	.16	17.7	.49	.24
17-1	58.5	22.6	8.5	2.3	1.08	.50	.28	.18	5.30	.56	.12
17-2	58.5	33.9	19.2	10.2	0.87	.48	.24	.09	1.94	.57	.15
17-3	58.5	19.8	11.3	(4.6)	1.55	.58	.37	.28	19.9	.80	.28
17-4	58.5	33.3	18.1	10.2	0.60	.30	.11	.02	1.12	.45	.09

0 = inlet; 1 = valve 1; 2 = valve 2; 3 = outlet

Table 6. Interpretation of the Test Data Using Model Calculations

Sample #	Inlet G+L (L/min)	Inlet [Fe^{2+}] (g/min)	Calculated [Fe^{2+}] and Differentials* (g/L)					
			Valve #1		Valve #2		Outlet	
			C	d	C	d	C	d
15-2	0.87	4.21	35.9	- 7.1	17.7	- 0.1	9.9	- 6.7
15-1	0.96	4.12	29.8	- 3.7	10.6	+ 2.5	2.6	- 0.6
15-3	1.41	6.73	25.3	+10.0	11.0	+ 8.6	5.3	+ 2.5
15-4	1.46	8.01	21.3	+14.0	9.2	+15.6	4.4	+ 7.4
16-3	0.99	4.87	38.8	-10.6	19.3	- 5.2	11.3	- 5.1
16-6	1.29	6.95	36.3	- 3.5	21.5	+ 2.2	15.7	+ 2.4
16-1	1.32	7.99	37.8	+ 2.9	24.8	- 1.1	19.7	- 2.2
16-5	1.41	6.95	30.7	- 1.9	15.7	- 1.6	9.8	- 4.2
16-2	1.59	7.99	29.0	+ 6.6	15.8	+ 1.1	10.7	- 2.2
16-4	1.81	9.73	29.2	+ 9.8	18.3	+ 3.2	14.1	+ 2.6
17-4	0.66	3.51	40.8	- 7.5	23.1	- 5.0	14.8	- 4.6
17-2	0.92	4.68	34.1	- 0.2	17.5	+ 1.7	10.5	- 0.3
17-1	1.16	4.68	23.7	- 1.1	5.8	+ 2.7	-	+ 2.3
17-3	1.68	7.31	17.7	+ 2.1	5.6	+ 5.7	0.8	+ 1.5

* see equation 3 and text; d = C(obs.) - C(calc.)

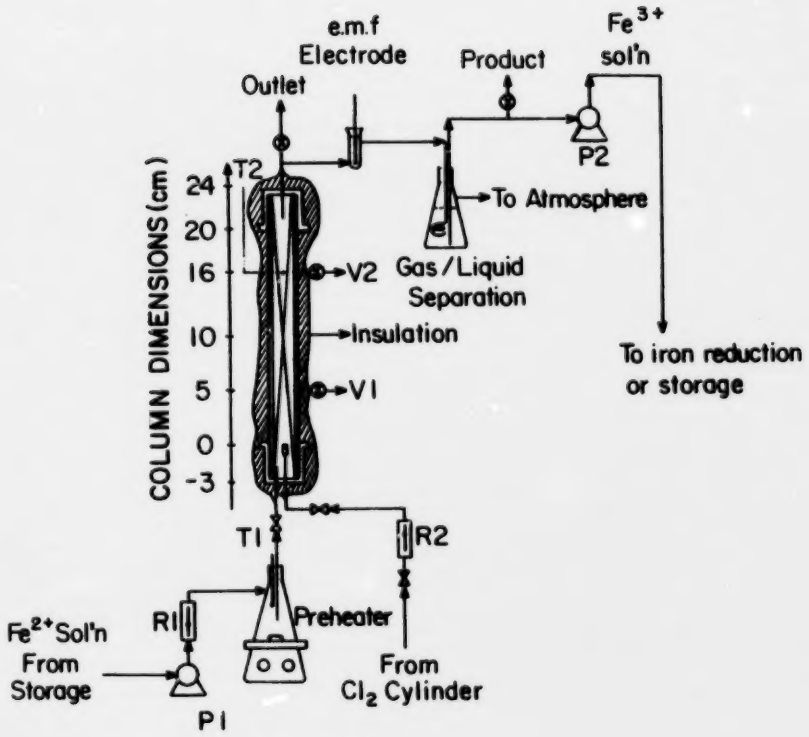


Fig. 1 - Experimental Set-up

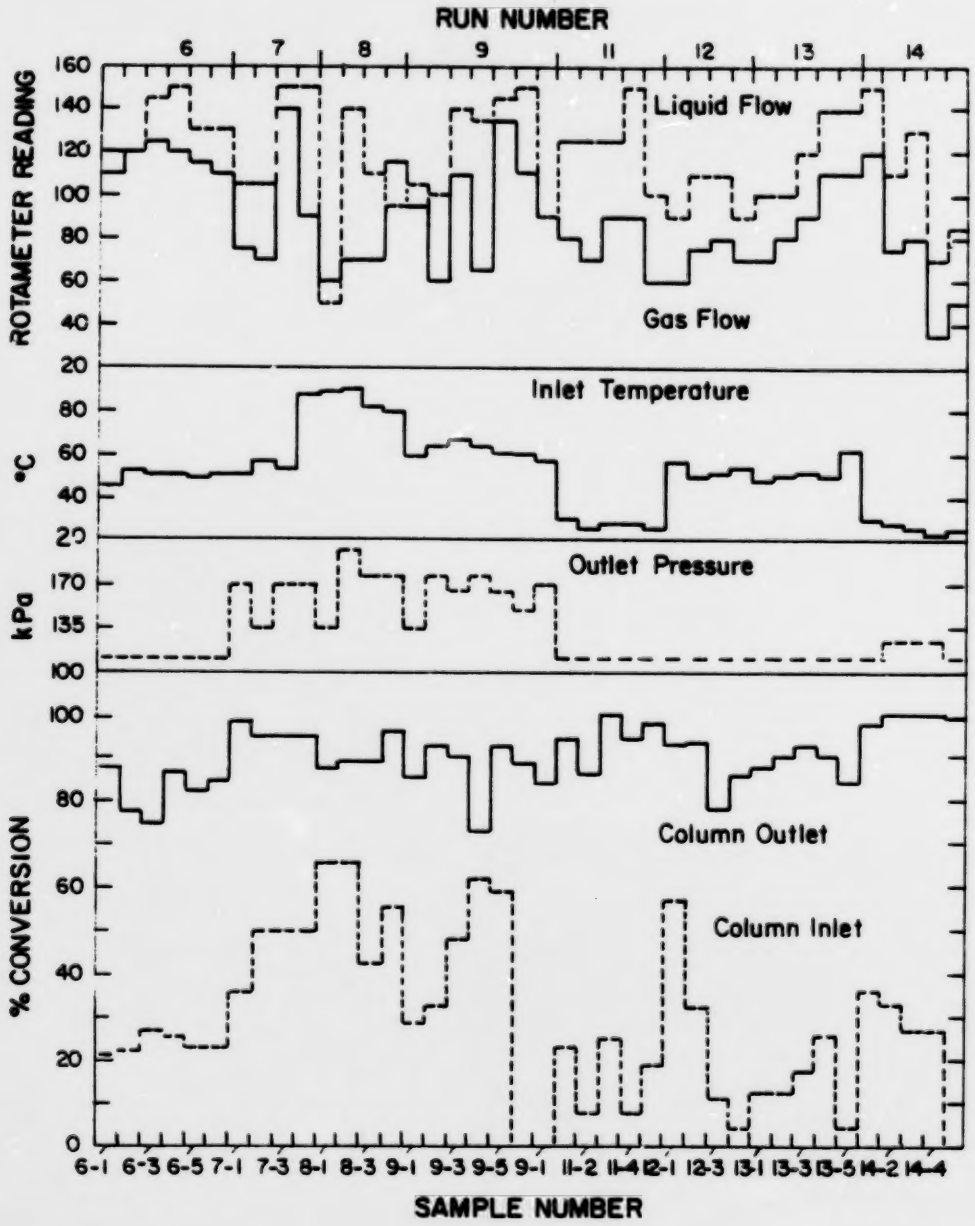


Fig. 2 - Summary of Preliminary Experiments

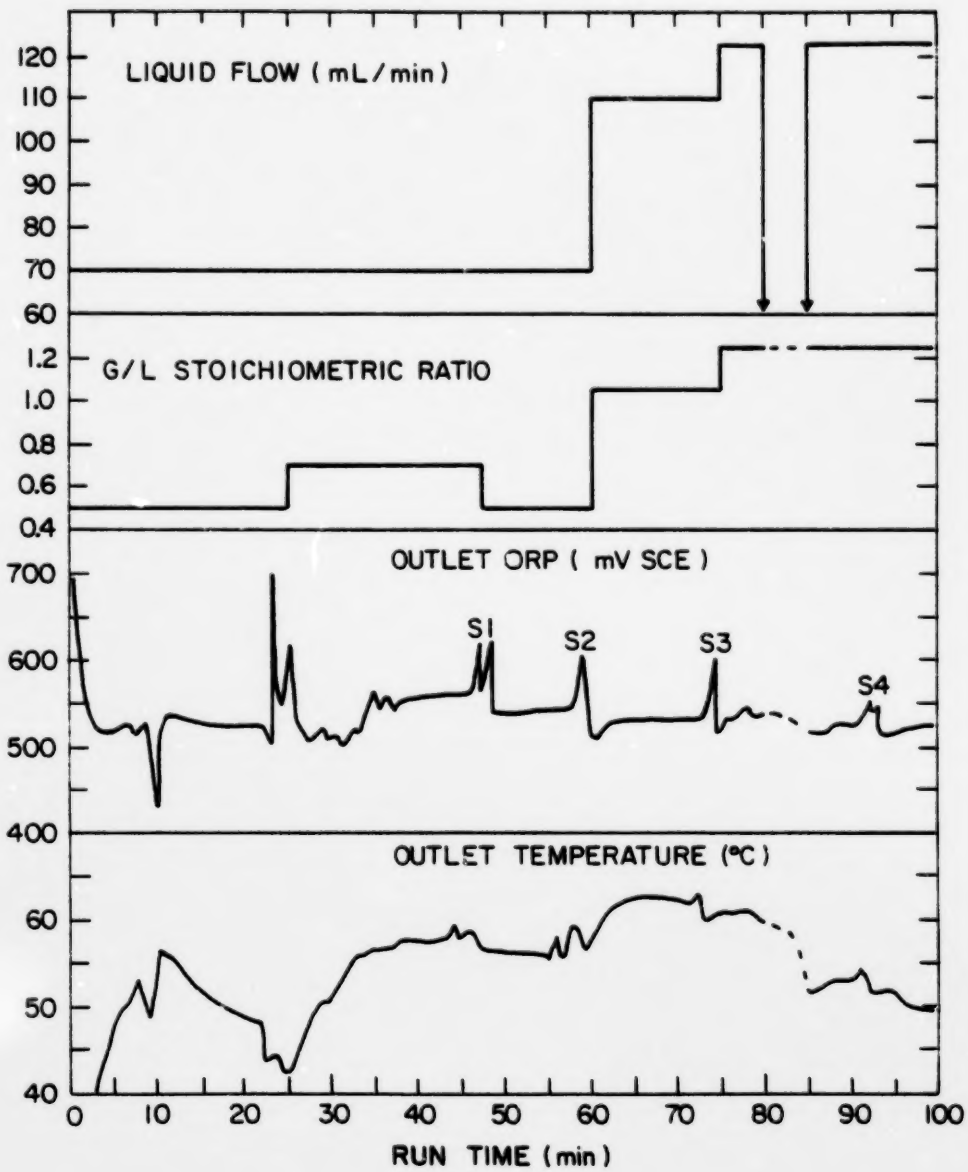


Fig. 3 - Summary of Test #15

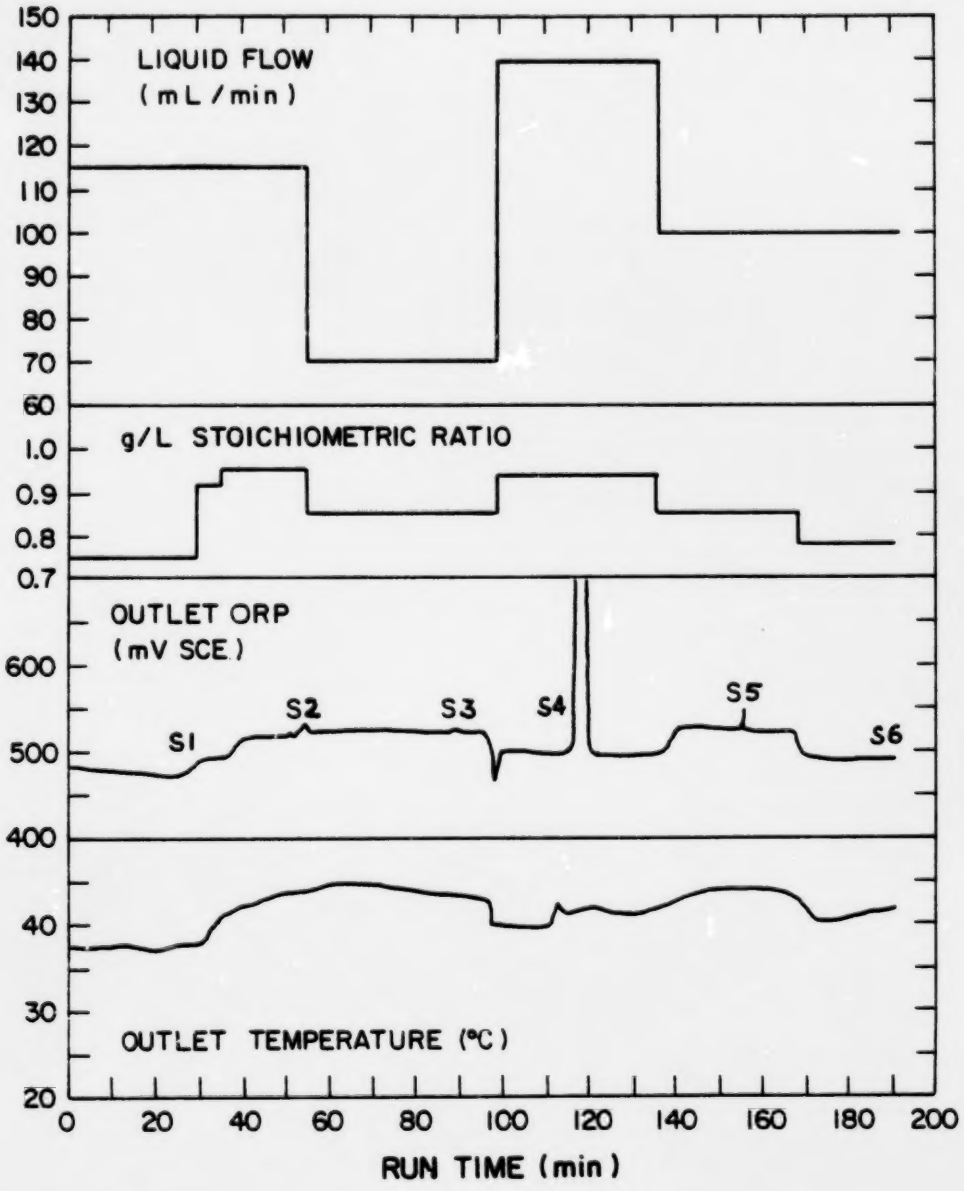


Fig. 4 - Summary of Test #16

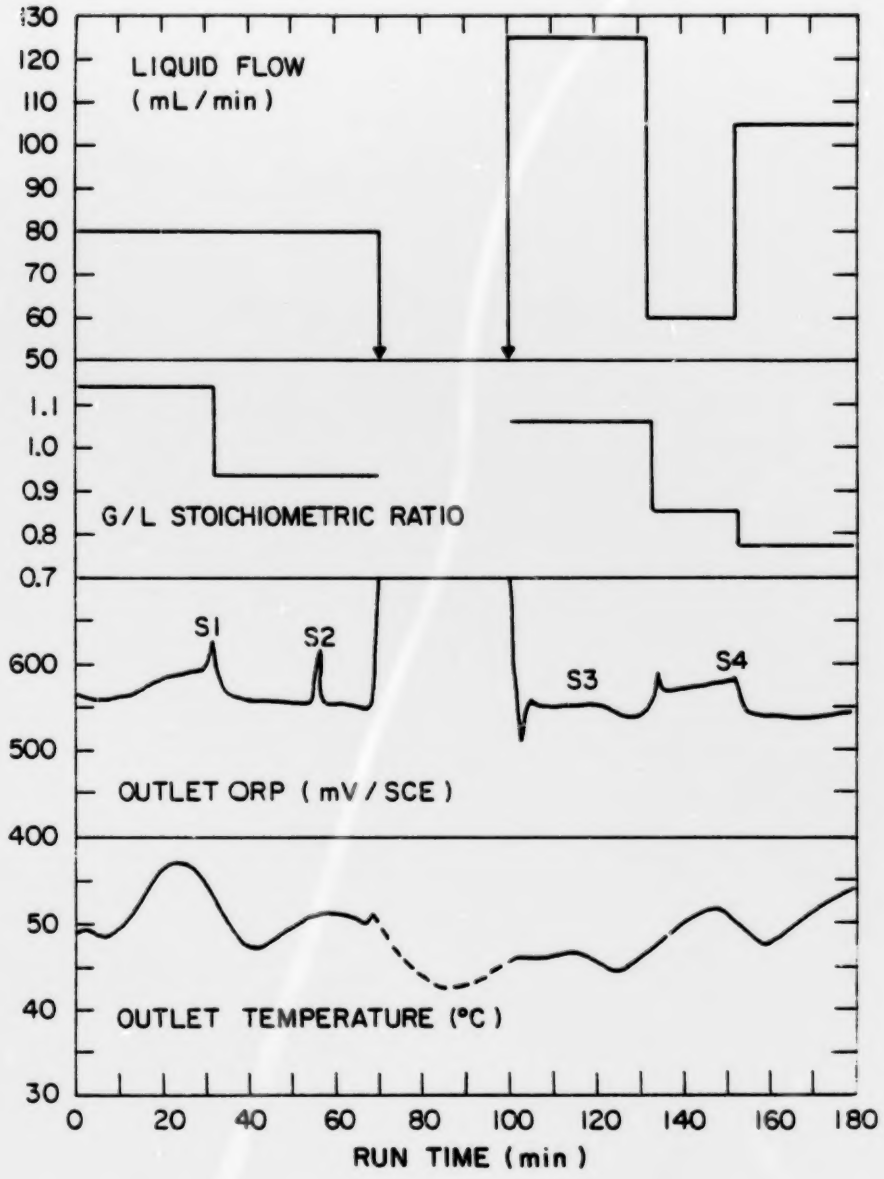


Fig. 5 - Summary of Test #17

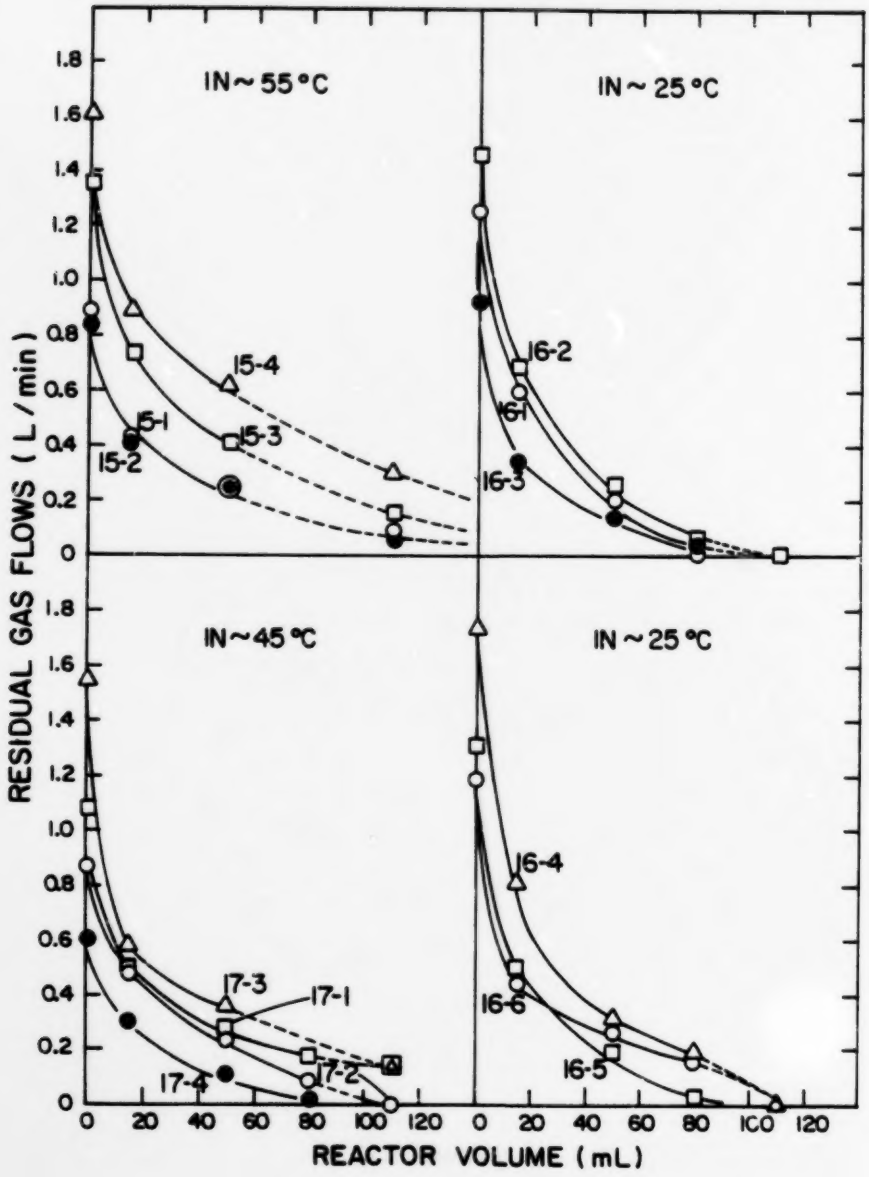


Fig. 6 - Measured Column Profiles

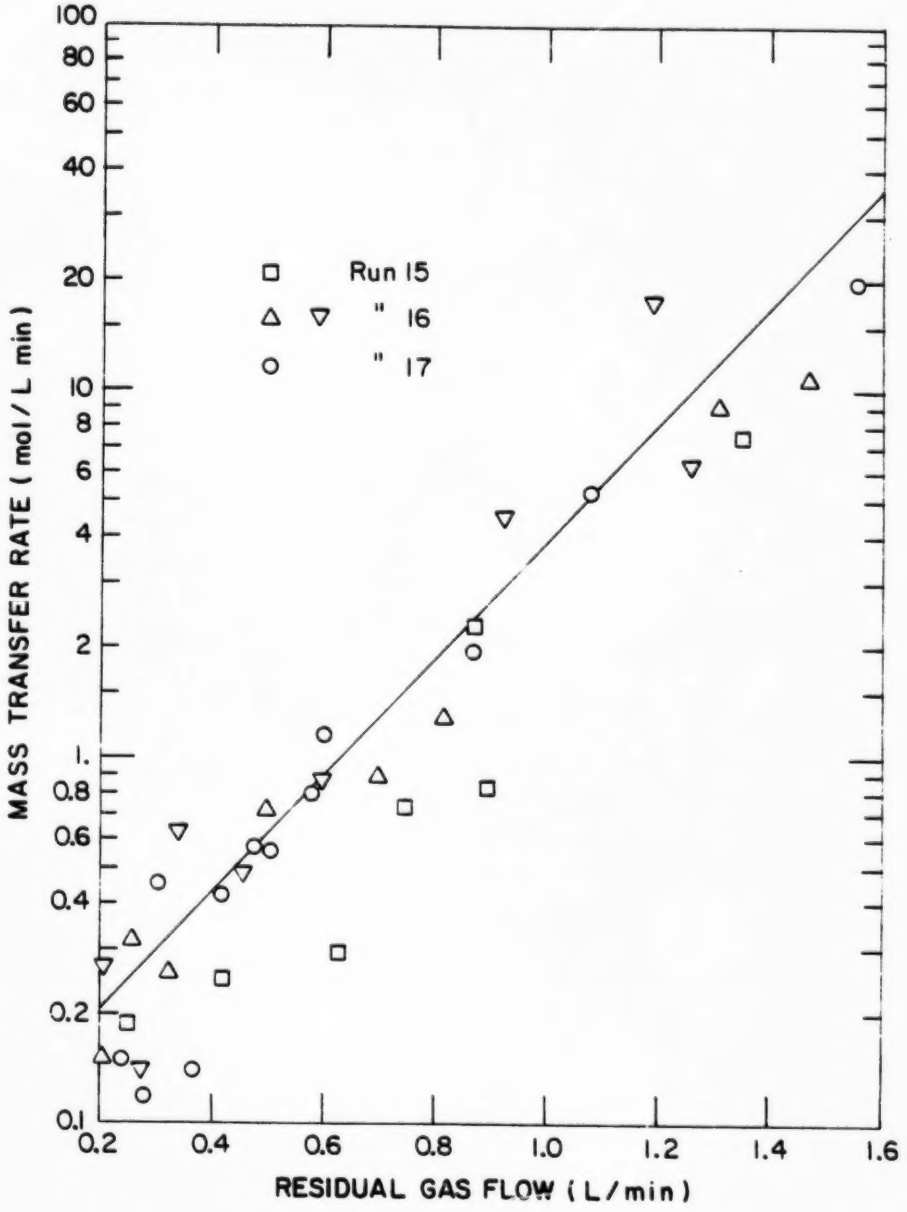


Fig. 7 - Mass Transfer Rate Dependence on the Residual Gas Flow